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Demonstration/Validation of Hazardous Air Pollutant-Free Torque Seal Inspection Lacquer

by Faye R. Toulan, Felicia Levine, John J. La Scala, Leslie Hasenbein, Genie Jones, and Ivan Davis

ARL-TR-4863 **July 2009**

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14. ABSTRACT
The U.S. Army uses numerous adhesives and sealants among other coating materials that contain significant amounts of hazardous air pollutants (HAP). The U.S. Army has determined that it is much more cost effective to reduce or eliminate HAP emissions from coatings operations rather than using emissions control devices.
This work examines laboratory and field demonstration/validation of one highly used sealant, Torque Seal. The adhesives and sealants thrust area of the Sustainable Painting Operations for the Total Army program has identified a HAP-free alternative to Torque Seal containing ethanol as the carrier solvent. Laboratory testing including adhesion, resistance to fluids, resistance to humidity, and drying time has validated that the HAP-free sealant performs very similarly to the baseline Torque Seal containing methanol (HAP). Furthermore, a demonstration/validation study at Fort Rucker, AL, using a UH-1 helicopter rotor shows that the HAP-free sealant has performed as well as the Torque Seal.

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sealant, HAP-free, adhesion, TGA, humidity, field trial, DSC

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1. Introduction

The U.S. Environmental Protection Agency (EPA) is planning to propose National Emission Standards for Hazardous Air Pollutants (NESHAP) for the surface coating of Defense Land Systems and Miscellaneous Equipment. The NESHAP is expected to require significant reductions in hazardous air pollutant (HAP) emissions from U.S. Army surface coating operations. Army installations are establishing environmental sustainability goals to exceed such regulatory requirements. The Sustainable Painting Operations for the Total Army (SPOTA) program has been established to manage the effort to reduce HAP emissions generated by Army coating operations. Adhesives and sealants are a small but significant portion of Army coatings HAP emissions.

Torque Seal, made by Organic Products Co. in Irving, TX, is a HAP-emitting sealant used in several Army applications. Torque Seal is used primarily to detect tampering or loosening of mechanical fasteners on military aircraft. Applied after bolts or fittings are in proper torque or position, this product gives inspectors visual evidence of any movement or tampering. Torque Seal dries to form a very brittle film that will crack, flake, or crumble when minimal force is applied. Other key product attributes include excellent adhesion to most surfaces and fast drying. The manufacturer refers to this product as an inspection seal lacquer or antisabotage lacquer. The fast-drying characteristic of Torque Seal is achieved by using low boiling point solvents as carriers, specifically ethanol and methanol. While both solvents are volatile organic compounds, only methanol is classified by the EPA as a HAP. The reported HAP content of this material is 20% by weight.

Torque Seal comes in a variety of colors (table 1), and all have the same essential properties. Although the total HAP emissions from Torque Seal are less than the emissions from many other surface coating operations, the product is used at more than 20 Army installations and accounts for ~5% of all Army sealant HAP emissions (*1*). Therefore, the SPOTA program focused on replacing Torque Seal with a similar product that contains no HAPs. After a thorough investigation, it was found that there was no similar product commercially available and no inspection lacquers of any kind that were HAP-free. As a result, a HAP-free version of the material was developed by the U.S. Army Research Laboratory (ARL) with Organic Products Co. containing only ethanol as the solvent. A single production batch of HAP-free Torque Seal was manufactured and used for both the lab testing and field trial. The vapor pressure of methanol and ethanol are 16.9 KPa and 7.87 KPa, respectively, at 25 ºC, and the vapor pressure is higher for methanol at all temperatures (*2*). The higher vapor pressure of the baseline sealant indicates higher volatility and will likely result in a faster drying time and, in this case, a higher tendency to crack.

Table 1. Quantity of Torque Seal used by Fort Rucker in 2003 (*1*).

National Stock Number (NSN)	Color	Number of 0.5-oz Tubes	Pounds of Material	Pounds of HAP
8030011432702	Red	1638	51.19	10.24
8030011250055	Orange	847	26.47	5.29
8030004081137	Green	42	1.31	0.26
8030011633483	Yellow	0	0.00	0.00
8030010777674	White	0	0.00	0.00
8030010668156	Blue	0	0.00	0.00
All NSNs	All	2527	78.97	15.79

This report summarizes both laboratory and field trial results for the new HAP-free formulation of Torque Seal containing only ethanol vs. the current product containing ethanol and methanol. The key properties discussed are rheology, nonvolatile content, thermogravimetric analysis (TGA), drying time (ASTM D 1640 [*3*]), adhesion (ASTM D 3359 [*4*]), resistance to fluid immersion (section 4.6.8 of MIL-PRF-85285D [*5*, *6*]), adhesion testing after fluid immersion, differential scanning calorimetry (DSC), flexibility, resistance to humidity (ASTM D 2247 [*7*]), and accelerated weathering (ASTM C 732 [*8*]). A field trial was conducted using a UH-1 helicopter at Cairns Army Air Field, Fort Rucker, AL, from May to November of 2008. Both formulations used in the field trial were supplied by the manufacturer Organic Products Co. in standard commercial packaging (0.5-oz plastic crimp tubes).

2. Experimental Method

2.1 Rheology

The viscosities of the wet sealants were measured using a TA Instruments (New Castle, DE) AR2000 Rheometer in steady shear flow experiments using a 40-mm, cross-hatched parallel plate geometry with a peltier and a solvent trap containing ethanol, at 20 °C. The purpose of the solvent trap was to keep samples from volatilizing during the experiment and skinning at the edges of the plate, resulting in drag or uneven flow. The shear rate was increased logarithmically from 10^{-5} s^{-1} to 1 s^{-1} and then decreased back to 10^{-5} s^{-1}, and 10 measurements were taken per decade. At a given shear rate, the shear stress was measured every 2 s. The shear rate and viscosity were recorded when the shear rate stabilized to within 5% tolerance for three consecutive intervals.

2.2 Nonvolatile Content (Solids)

Wet Torque Seal samples were completely evaporated to determine the solid and volatile content in the sealant. An aluminum weighing pan was tared, and ~5 g of thoroughly mixed sealant was poured into the tared container, covered, and weighed. After removing the cover, the container was placed in an oven at 70 °C ± 1.1 °C (158 °F ± 2 °F) until the sample reached a constant weight. The covered container with the sample was cooled to 23 C° ± 1.1 °C (73.5 °F ± 2 °F) before weighing. Each sample was run in duplicate. The percentage of total solids was calculated as follows:

$$\text{Total solids (\%)} = (\text{weight of residue/original weight of sample}) \times 100. \tag{1}$$

2.3 Thermogravimetric Analysis (TGA)

TGA was run on the samples of dried formulation solids using a TA Instruments TGA 2950. Wet formulation samples were dried in an oven until the sample weight remained constant. This dried material was pulverized in a coffee bean grinder to allow the release of any trapped solvent. Oven drying of the pulverized sample continued until the sample weight was again constant. A 5- to 10-mg sample of dried, pulverized resin and pigment was placed on a tared platinum sample holder suspended on a weight-sensing wire hook. The samples were run in air up to 800 °C at 10 °C/min. The TGA instrument measures the sample mass as a function of temperature throughout the experiment. The percent mass remaining at the end of the run is the residual inorganic ash content. Knowing the percentage of ash in the nonvolatile portion of the formulation can allow an estimation of the pigment to binder ratio (P/B). The organic resin, which acts as the binder, will be burned off in TGA analysis, and the remaining ash will be comprised of the pigments, extenders, and other fillers in the formula. P/B can be a useful parameter for formula comparison. Three samples of each formulation were run to get a good measure of percent error.

2.4 Drying Time ASTM D 1640

Beads of the sealants were applied on a glass plate and evaluated for skin time at room temperature. Skin time (also known as open time) is the formation of a cohesive film that can withstand a light touch with a wooden dowel (*9*). The product was applied to a uniform thickness of 6.5 mil. The tack-free time of the samples was measured periodically with a common qualitative industry “touch-test” until the samples were no longer tacky and resisted transfer. Lastly, tack-free time of the samples was evaluated using cotton fibers. Testing tack-free time with cotton fibers is a more sensitive touch-test that determines the ability of the sample to resist adhesion to the fibers. When the fibers do not transfer to the sealant and the film does not deform, the product is dry or tack-free. The time required for the sample to become tack-free was recorded as a range rather than a single data point. Drying time studies were performed at –11, 75, and 140 ºF as outlined in the demonstration/validation plan for Torque Seal. A typical average temperature for application of the sealant at a depot is 75 ºF, whereas

–11 ºF and 140 ºF encompass the lower and higher ends of the reasonable temperature application spectrum.

2.5 Adhesion Testing ASTM D 3359

The baseline and HAP-free sealants were applied to a uniform thickness of 6.5 mil on steel substrates that measured 4 × 6 in. Conditioning parameters for adhesion testing were –11, 75, and 140 ºF. The sealants were allowed to dry for 24 h at room temperature and conditioned for 2 h before adhesion testing using Test Method B cross-cut tape test. Tape was applied to the cross-hatched area and then removed. The amount of sealant remaining in the cross-hatched area was rated according to ASTM D 3359 to assess the adhesion performance of the sealants (*10*).

2.6 Resistance to Fluid Immersion Section 4.6.8 of MIL-PRF-85285D

The baseline and HAP-free sealants were applied to a uniform thickness of 6.5 mil on steel substrates that measured 4 × 6 in and allowed to dry 24 h at room temperature. Dried films were challenged by standing the panels vertically in a container of fluid so that about half of the film area was immersed. The following liquids and test conditions were used:

- MIL-L-23699 (*5*) lubricating oil at 121 °C for 24 h.
- MIL-PRF-83282 (*5*) hydraulic fluid at 66 °C for 24 h.
- JP-8 fuel at room temperature for 7 days.

The films were examined 1 h after removal from the fluid for blistering, delamination, and discoloration.

2.7 Adhesion Testing Conducted on Fluid Immersion Samples

Approximately 2 weeks following the completion of the fluid immersion evaluation (as described in section 2.6), adhesion testing at 75 °F was conducted (section 2.5) on the portion of sealant submerged in the specified fluids (*4*, *5*).

2.8 Differential Scanning Calorimetry (DSC)

DSC calculates the heat flow into or out of a material by measuring the temperature difference between a sample pan and an empty reference pan being heated under identical conditions. The glass transition (T_g) of a polymer from a rigid to a rubbery material can be seen as an inflection point on the ascending temperature DSC curve. Samples of the baseline and HAP-free material were dried and pulverized before analysis. A small sample (5–10 mg) was crimped in a pan and placed on one sensor in the sample chamber while an empty reference pan was placed on the other sensor. The chamber was sealed and heated under nitrogen, and temperature differences between the sample and the reference were measured. The samples were exposed to two heat cycles, from –10 to 200 °C and back to –10 °C at a rate of 10 °C/min.

2.9 Flexibility Testing

Flexibility testing was performed using the mandrel bend test. The sealants were applied to a uniform thickness of 6.5 mil on tin-coated steel Q-panel substrates that measured 3 × 6 in. The samples were allowed to dry for 24 h (at room temperature) then conditioned at one of three temperatures (–11, 75, and 140 ºF) for 2 h prior to flexibility testing on a 1/8-in mandrel. The conditioning time at the specified temperatures was increased until cracking occurred at all three temperatures. The Q-panels were bent along the sides of a cylindrical mandrel, with the sealant on the opposite side of the mandrel to provide a flexure stress. Because Torque Seal is designed to crack when tampered with, this sealant breaks at low flexural stresses. The baseline and HAP-free sealants were characterized for "crack time" occurring at each of these temperatures.

2.10 Humidity Testing ASTM D 2247

The baseline and HAP-free sealants were applied to a uniform thickness of 6.5 mil on steel substrates that measured 4 × 6 in. The sealants were allowed to dry for 3 days. Prior to humidity exposure, the bare steel portion of the panels was covered with a clear packing tape to prevent corrosion. The specimens were then subjected to constant conditions of 40 °C and 100% relative humidity. Samples were evaluated after 1, 3, 7, 14, and 30 days of exposure for discoloration, blistering, and delamination.

2.11 Accelerated Weathering ASTM C 732

The baseline and HAP-free sealants were applied to a uniform thickness of 6.5 mil on steel substrates that measured 3 × 6 in. The sealants were allowed to dry for 3 days before starting accelerated UV exposure in a QUV[*] accelerated weathering tester cabinet. Torque Seal samples were exposed to a 340-nm UVA light source at a radiance level of 0.77 W/m^2 for 8 h at 60 ºC followed by 4 h of condensation at 50 ºC without light exposure. Samples were examined at 100 and 500 h of exposure for discoloration, blistering, and delamination.

2.12 Demonstration/Validation Plan

ARL and Fort Rucker conducted a field trial on 6 May 2008 at Cairns Army Air Field, Fort Rucker, AL. Both the baseline and HAP-free Torque Seal products were applied to the UH-1 helicopter rotor system (figure 1) on approximately 40 assemblies of nuts, bolts, and studs. On the day of application, a decision was made to place a bead of each sealant on every fastener, as seen in figure 2, for a more accurate side-by-side evaluation. In addition, the operator was required to photograph the sealant on each fastener to document any degradation of the sealant as a function of time. Photos were also taken at 7, 14, 30, 60, 90, and 120 days after application. Inspections were conducted at these same time intervals for delamination (peeling away from surface), deterioration, and operation-induced cracking (*11*). The UH-1 helicopter participated in regular training operations under representative flight conditions. This ensured that the HAP-

[*]QUV is a registered trademark of Q-Panel Lab Products, Cleveland, OH.

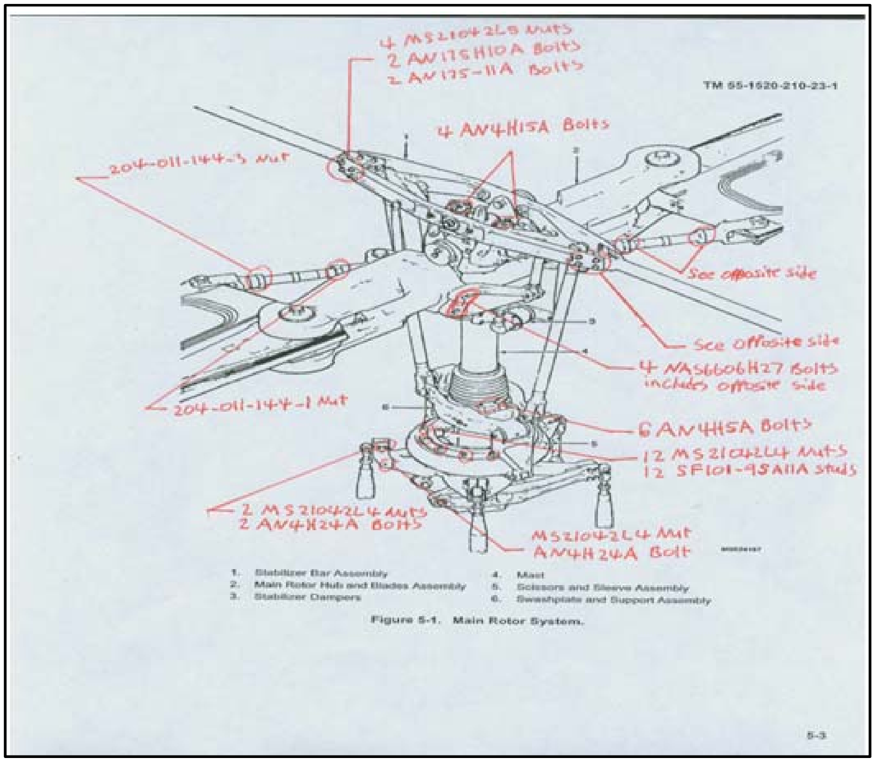

Figure 1. UH-1 helicopter rotor system diagram.

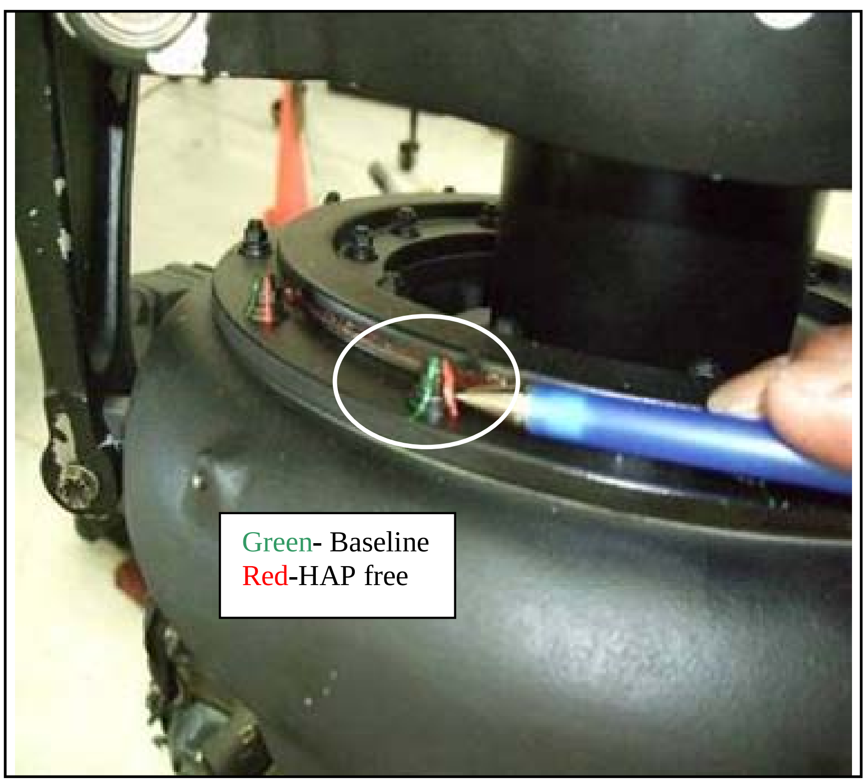

Figure 2. Torque Seal applied to UH-1 helicopter.

free and baseline Torque Seal formulations were subjected to vibrations and stresses sufficient to demonstrate their performance. This test is important to prevent the occurrence of false tampering incidents.

3. Results and Discussion

3.1 Rheology

Both of the sealants tested are non-Newtonian shear thinning fluids, thus their viscosities change with shear rate. Typically, there is a Newtonian plateau at very low shear rates where viscosity is independent of shear rate before the commencement of shear thinning behavior. The power law region of the shear thinning curve has a viscosity as follows:

$$\eta = \kappa\gamma^{\,n-1}, \qquad (2)$$

where η is the viscosity, κ is the flow consistency index, γ is the shear rate, and n is the flow behavior index. The values of κ and η were calculated and used to characterize each product formulation (table 2). The baseline product was more viscous than the HAP-free product, as seen in figure 3. Despite using a solvent trap to minimize skinning of the product at the edges of the rotating plate, this higher Newtonian viscosity may be a result of the higher volatility of methanol. However, the shear thinning behavior of both products was similar.

Table 2. Rheology data for Torque Seal.

Torque Seal Product	**Newtonian Viscosity (Pa*s)**	**Standard Deviation**	**"κ" Flow Consistency Index**	**"n" Flow Behavior Index**	**R^2**
Baseline	25,800	878	235	0.4687	0.9944
HAP-free	11,587	208	37	0.3275	0.9989

3.2 Nonvolatile Content (Solids)

The average calculated solids by weight of the baseline (51% ± 0.53) and HAP-free Torque Seal (54% ± 0.46) were similar. The slight difference between the two products is attributed to the higher evaporation rate of methanol as compared to ethanol. It is possible that the HAP-free formula trapped some slower evaporating ethanol in the dried samples. According to the manufacturer of Torque Seal, the theoretical solids of this product are 50% by weight with the other 50% by weight composed of volatiles. These results are not unusual for commercial products where there are normal variations in manufacturing (including weighing errors) and packing operations.

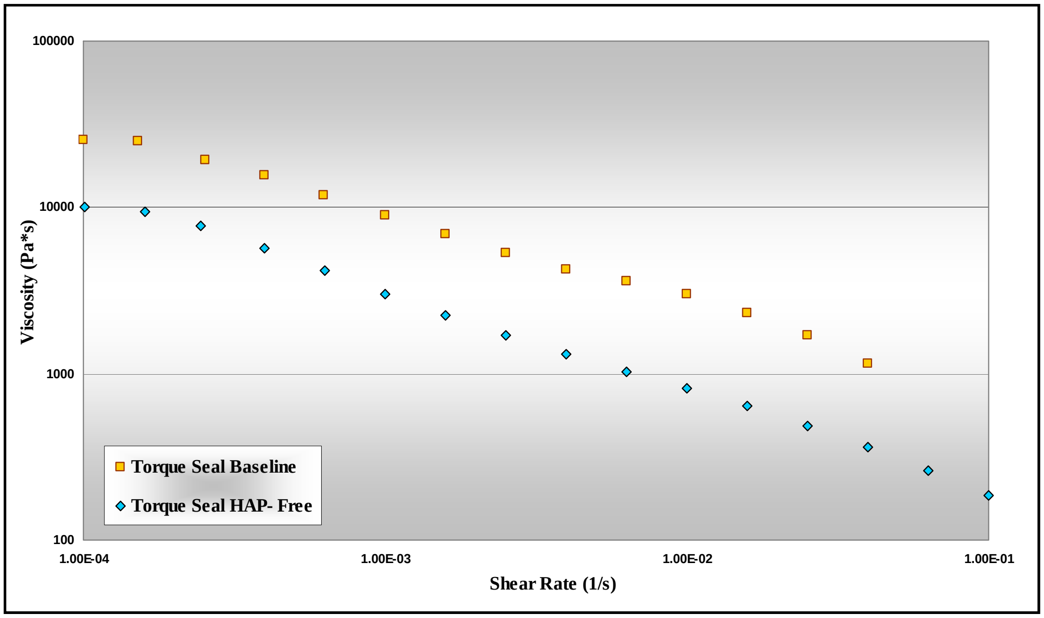

Figure 3. Torque Seal rheology.

3.3 Thermogravimetric Analysis (TGA)

The solids remaining after volatile evaporation (dry solids) were tested by TGA. The percentages of organic polymer binder and inorganic ash content represent the solid phase of the Torque Seal. Both sealants were relatively similar in organic and inorganic content (±3%). This is an acceptable variance and can be attributed to inconsistency of the manufacturing process of the product and experimental error in the laboratory analysis (table 3).

Table 3. TGA (dry) calculated results.

Torque Seal Product	Solids in Wet Product (%)	Organic Binder in Solids (%)	Inorganic Ash in Solids (%)
Baseline	51	73	27
HAP-free	54	76	24

The temperatures at which thermal degradation occurs (indicated by sharp sample weight loss) can give some insight to the types of chemical bonds within those materials. Figure 4 illustrates the thermal degradation of the organic binder. The remaining weight fraction above 600 °C is inorganic ash, which is not subject to oxidative degradation at these temperatures. Materials that comprise the ash portion of a formulation potentially include inorganic pigments, clay thickeners, mica, and other mineral ingredients act as fillers and rheology modifiers.

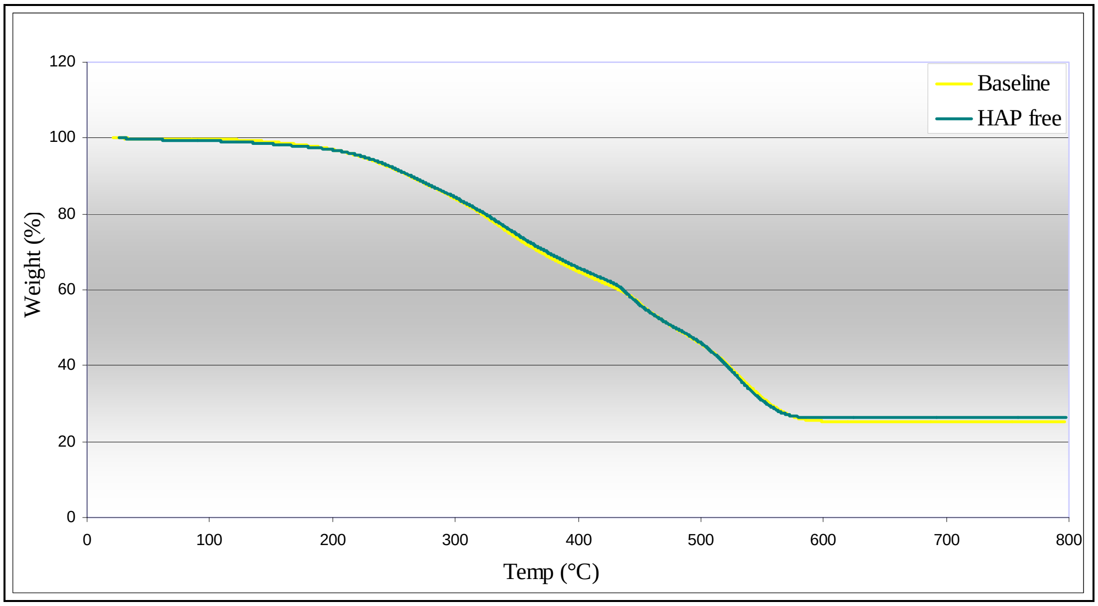

Figure 4. Torque Seal TGA of dry solids.

The TGA derivative curve (figure 5) gives a better indication of decomposition temperatures. The peaks correspond to degradation temperatures of particular chemical bonds. Without running a more probative test to discern exact chemical composition, it is likely that the large peak above 500 °C is representative of the C-H bond degradation, the narrower peak between 400 and 500 °C is most likely the C-C bond cleavage, and the broader peaks below 400 °C are most likely from the C-O bonds. The more important point is the baseline and HAP-free products have similar degradation profiles. This result was expected since the carrier solvent was the only different ingredient and the solid formula ingredients were presumed to be the same.

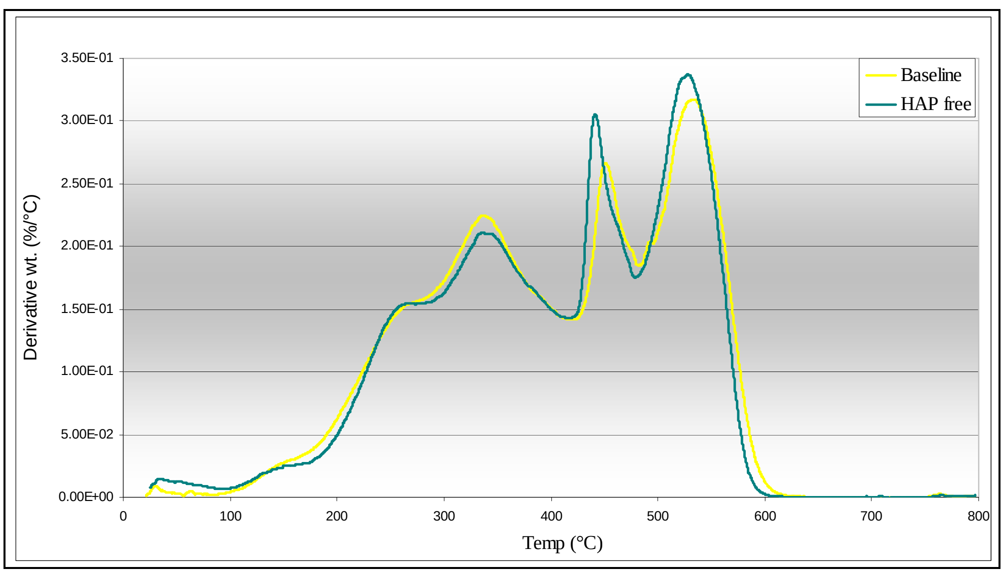

Figure 5. Torque Seal TGA of dry derivative weight.

3.4 Drying Time ASTM D 1640

All the values in table 4 were recorded as a range of time as these tests are qualitative in nature. Drying time studies were performed at –11, 75, and 140 ºF. In every leg of this test method, the drying time was slightly longer for the HAP-free sealant relative to the baseline. This result was expected because ethanol has a lower volatility than methanol. The difference in drying time was not noticeable by the user during application.

Table 4. Drying time results for Torque Seal.

Test Temperature (°F)	Baseline			HAP-Free		
	Skin on Bead (min)	Tack-Free by Touch (min)	Tack-Free by Cotton (min)	Skin on Bead (min)	Tack-Free by Touch (min)	Tack-Free by Cotton (min)
–11	27–29	37–39	38–40	33–35	70–72	71–73
75	10–12	19–21	20–22	13–15	23–25	24–26
140	5–7	6–8	8–10	7–9	9–11	10–12

3.5 Adhesion Testing ASTM D 3359

The green sample is the baseline Torque Seal containing HAPs, while the red sample is HAP-free (figure 6). Adhesion testing was conducted at three different temperatures to simulate various service conditions the sealant may encounter during use (–11, 75, and 140 °F). The HAP-free product performed similarly to the baseline product at all three temperature settings. Results for adhesion at –11 °F (figure 6) and 140 °F (not shown) had a rating of 4B, meaning small flakes of the coating are detached at intersections and less than 5% of the area is affected (*4*). Adhesion performance at 75 °F (not shown) had a rating of 5B, meaning the edges of the cuts are completely smooth and none of the squares of the lattice is detached. A rating of 5B is the best that could be given by ASTM D 3359. According to the test method, both 4B and 5B ratings are considered passing results.

3.6 Resistance to Fluid Immersion MIL-PRF-85285D

The films were examined 1 h after removal from the fluid for blistering, delamination, and discoloration. Both the baseline sealant (green) and the HAP-free sealant (red) showed a darkening of color after immersion for 24 h in lubricating oil at 121 °C (figure 7), while no blistering or delamination occurred. The darkened color observed below the immersion line was likely due to oil absorption into the film. Maximum color change appeared to occur in the first 24 h of immersion and remained somewhat constant after that. However, both the baseline sealant and the HAP-free sealant showed no change after immersion for 24 h in hydraulic fluid at 66 °C (not shown). Similarly, both the baseline sealant and the HAP-free sealant showed no change after immersion in JP-8 fuel at room temperature for 7 days (not shown).

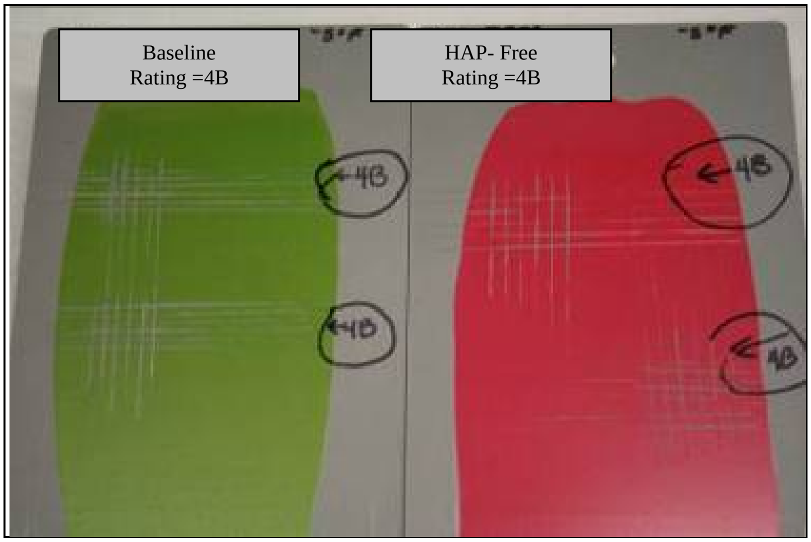

Figure 6. Cross-cut adhesion tape test at –11 °F.

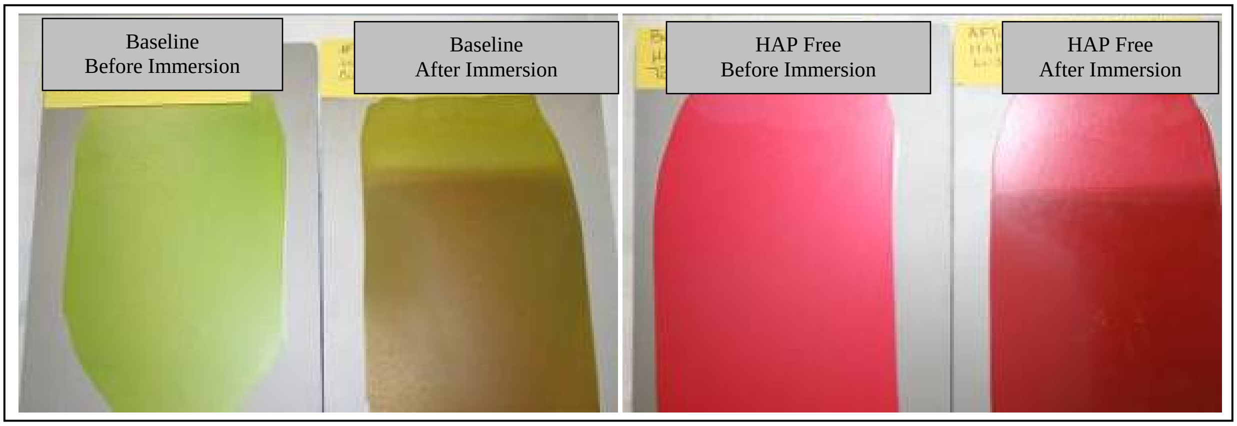

Figure 7. Immersion in lubricating oil at 121 °C for 24 h.

3.7 Adhesion Testing Conducted on Fluid Immersion Samples

The HAP-free sealant (red) performed similarly to the baseline sealant (green) and had good ratings across the board. The adhesion rating after 24-h immersion in lubricating oil at 121 °C was 5B (not shown). Chemical or physical alterations from fluid immersion (oil absorption, component leaching, film softening, etc.) had no effect on adhesion of the sealants to the substrate. The adhesion rating after 24-h immersion in hydraulic fluid at 66 °C (not shown) and after 7 days in JP-8 fuel at room temperature was 4B (figure 8). Therefore, although immersion in these fluids caused no visible effects, it is apparent that immersion reduced adhesion to the substrate slightly vs. the portion of the film not immersed in fluid.

Figure 8. Adhesion after 7-day immersion in JP-8 fuel at room temperature.

3.8 Differential Scanning Calorimetry (DSC)

Torque Seal is designed to be brittle. A reduction in T_g would likely reduce the brittleness of this material, making it less susceptible to cracking and therefore a less effective inspection lacquer. A graph of heat flow (milliwatts) vs. temperature was obtained and an approximate T_g temperature of 73 °F (figure 9) was determined for both the baseline and HAP-free product. Therefore, we can conclude that the replacement of methanol with ethanol had no effect on the ultimate T_g of this sealant.

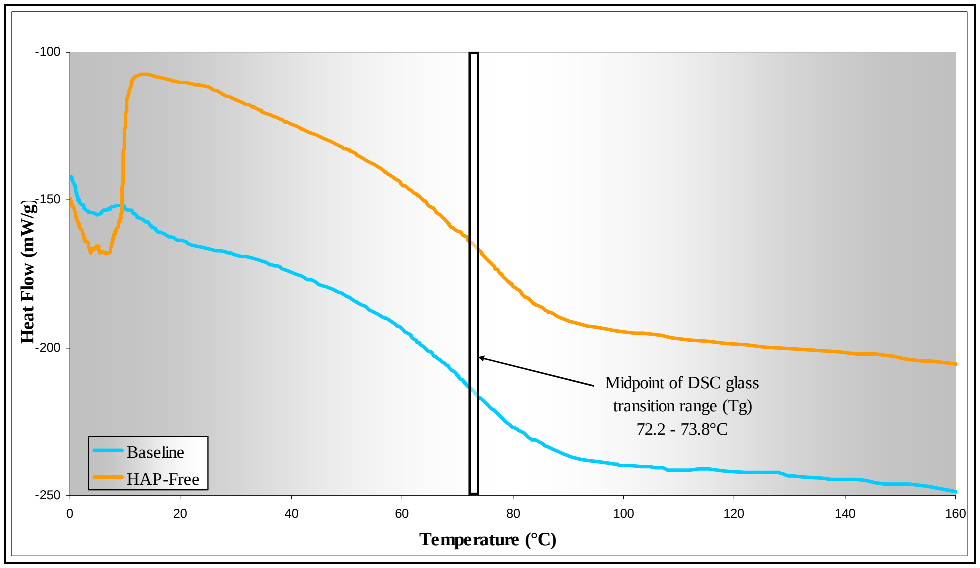

Figure 9. Torque Seal DSC.

3.9 Flexibility Testing

Since Torque Seal is designed to crack when tampered with, this sealant breaks at low flexural stresses. Flexibility was tested at –11 °F, room temperature, and 140 °F. The conditioning temperature is an important factor in determining the time it takes for the sealants to cure. After 44 days of conditioning at –11 °F, neither the baseline nor the HAP-free sealants showed any stress fractures on the mandrel bend test (not shown). This is likely due to incomplete evaporation of the solvents in the sealant at this low temperature. The solvent remaining in the applied film acts as a plasticizer and keeps the sealant flexible. Both room temperature samples showed stress fractures after 12 days of conditioning (not shown). Additionally, both the baseline and HAP-free samples showed stress fractures at the site of the bend after conditioning at 140 °F for 4 h (figure 10). The baseline and HAP-free products performed similarly to each other for all three temperature parameters.

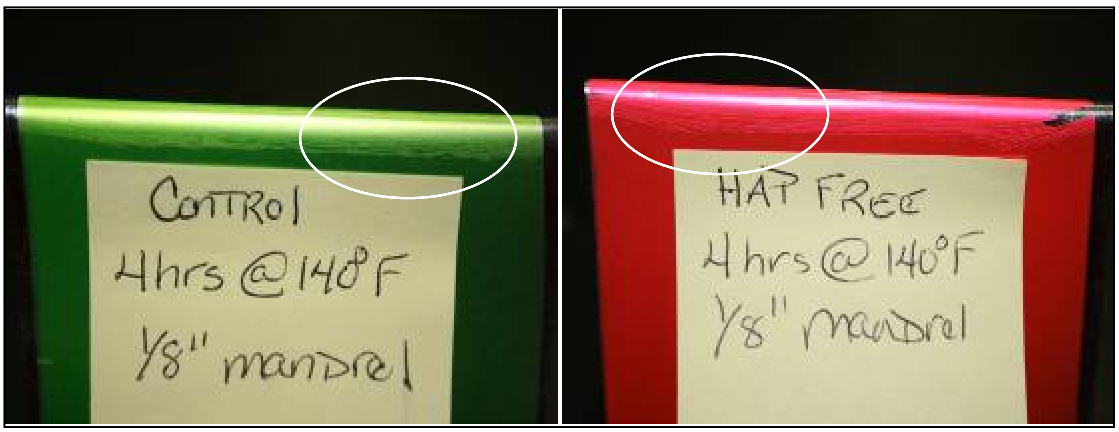

Figure 10. Torque Seal mandrel bend test at 140 °F.

3.10 Humidity Testing ASTM D 2247

No delamination (the failure of the adhesive, either in the adhesive itself or at the interface between the adhesive and the adherend) or blistering (*9*) occurred on any of the panels. However, fading in color to a similar extent for both the baseline and HAP-free sealants was visible. The samples exposed for 1 day experienced a dramatic color fade. Continuing exposure for up to 4 weeks did not show a greater degree of color fading than the 1-day exposure (figure 11). Any uneven fading can be attributed to the position of the moisture spray nozzle inside the humidity cabinet. This phenomenon was verified by repositioning the various sample panels to duplicate the effect. It is apparent that the pigments used in both the baseline and HAP-free sealants were extracted by the condensate on the sealant surface. Note that the baseline and HAP-free formulations behaved the same under humidity testing.

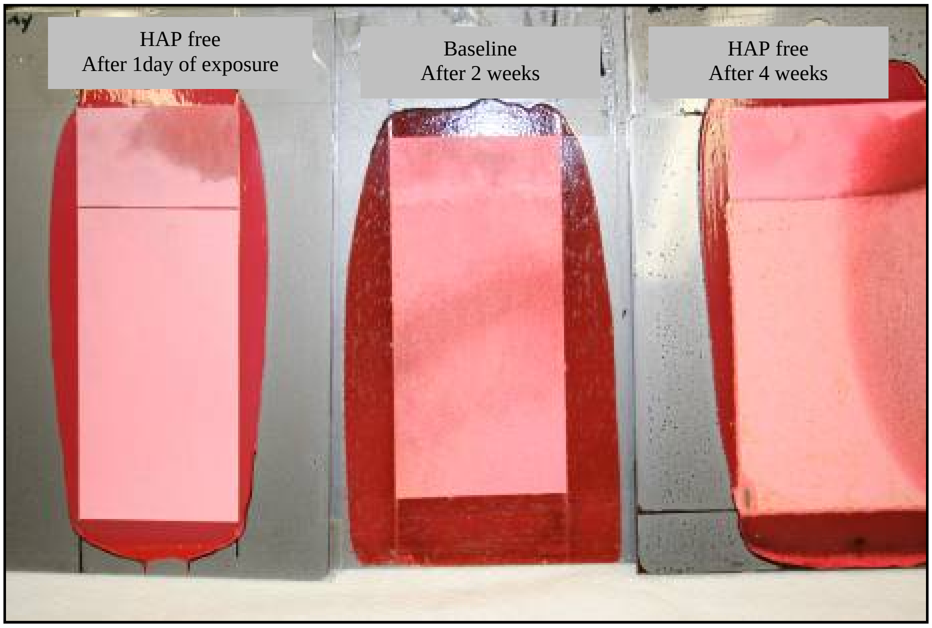

Figure 11. Color change after exposure to 100% humidity and 40 °C.

3.11 Accelerated Weathering ASTM C 732

After 100 h of exposure, both the baseline and HAP-free samples faded considerably; thus, gloss was reduced from ~40 units to ~2 units at an observation angle of 60°. In addition to a reduction in gloss, figure 12 shows extensive chalking. Chalking is defined as the formation of powder, usually white in color, on the surface of a sealant caused by the disintegration of the polymer or binding medium due to weathering (*9*). After 500 h of exposure, the gloss and color remained the same as after 100 h; however, the samples cracked and delaminated from the substrate (figure 12). Again, the HAP-free sample performed comparably to the baseline sample.

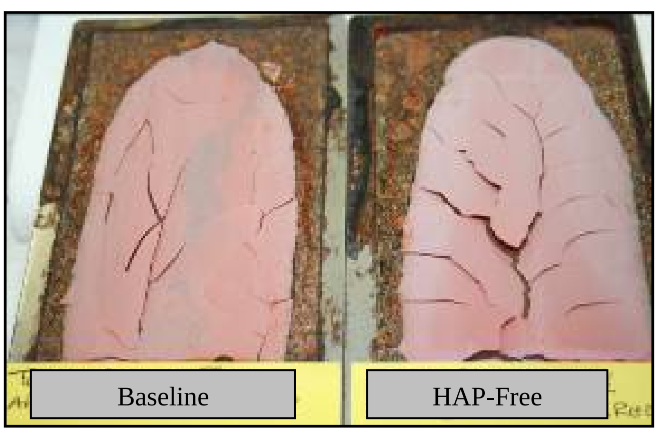

Figure 12. Torque Seal QUV results after 500 h of exposure.

4. Field Trial on UH-1 Helicopter at Fort Rucker

The HAP-free Torque Seal formulation and the commercially available (baseline) Torque Seal were applied side-by-side to a UH-1 helicopter (ID no. 0-16394) rotor system. The operator commented that there was no perceptible difference in ease of application (spreadability, viscosity, runniness, etc.) or drying time between the two sealants. The sealants were examined for delamination (peeling away from surface) and operation-induced cracking after 7, 14, 30, 60, and 90 days. Checklists (found in tables 3–11 of the demonstration/validation plan [*11*]) were used during these examination periods of the field trial to document and consistently communicate observations. Originally, the test duration was scheduled for 120 days, but the Fort Rucker team ended the demonstration at 90 days, maintaining that the HAP-free sealant was comparable to the baseline. The team at Fort Rucker made the following conclusions /recommendations upon completion of the field trial:

> In our opinion, the performance of the HAP-free formulations and current Torque Sealant are similar with respect to delaminating and cracking. Both sealants adhere to clean nongreasy surfaces longer than on greasy surfaces. As for cracking, both brands of sealants displayed similar characteristics, cracking slightly within 60 days then more over time as they aged, eventually maturing to a state of no additional cracks. In conclusion, when focusing on cracking and delamination, both the traditional and HAP-free torque sealant performed equally well with neither brand performing better than the other. We would recommend substitution of the HAP-free formulation, which contains 50% ethanol and 50% solids (eliminating methanol), as a viable alternative to assist in meeting NESHAP requirements while continuing to meet mission expectations.

Table 5 shows a summary of the field test data as recorded by the UH-1 service technicians. If a bead was recorded as cracked or delaminated at the 30-day check, it was not counted again in the 90- and 120-day checks. In other words, each check only recorded the new cracks or delamination since the previous inspection. However, if a bead was cracked at the 30-day check and delaminated (in part or whole) at the 90-day check, it would be recorded once for each occurrence. Figure 13 illustrates a partial delamination of the baseline sealant (green) located at the base of the nut/bolt assembly. Similar but more subtle is a partial delamination of the HAP-free sealant (red) also located at the base of the nut/bolt assembly. The higher number of cracked and delaminated sealant beads for the baseline sealant relative to the HAP-free sealant was likely due to the higher volatility of methanol, resulting in lower remaining solvent content within the solid bead. Although the total number of cracked and delaminated baseline beads was slightly higher than the number of HAP-free beads, the overall performance characteristics of the two sealants were qualitatively similar. The majority of the cracking and delamination occurred at the 60-day interval, at which point the sealants appeared to perform comparably.

Table 5. Fort Rucker summary of field trial results.

Inspection Time After Application	Number of Cracked Sealant Beads		Number of Delaminated Sealant Beads	
	Baseline	HAP-Free	Baseline	HAP-Free
7 days	3	1	0	0
14 days	2	2	0	0
30 days	3	2	1	0
60 days	24	13	16	13
90 days	4	9	16	11
Totals	36	27	33	24

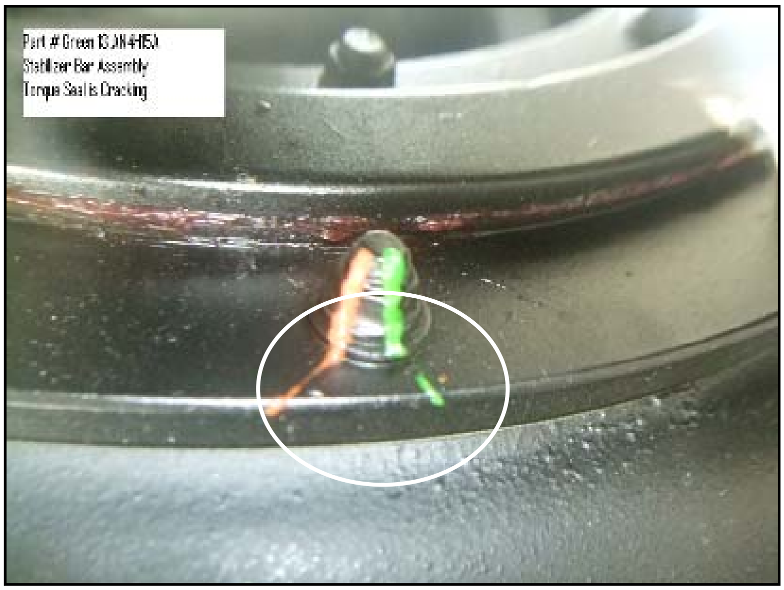

Figure 13. Torque Seal 60 days after application to a UH-1 helicopter.

5. Conclusions

Laboratory testing indicated that the HAP-free Torque Seal using ethanol as the lone solvent performed similarly to the baseline product containing both methanol (HAP) and ethanol. Results were consistent across the complete test series. Testing showed little to no difference in rheology, drying time, adhesion, and weathering/exposure for the HAP-free sealant relative to the baseline product. Furthermore, a demonstration/validation study at Fort Rucker, AL, on a UH-1 helicopter showed that the HAP-free sealant performed suitably as a substitute material. Replacing the baseline Torque Seal with the HAP-free version will reduce HAP emissions at more than 20 installations across the Army. ARL and Fort Rucker recommended the replacement of the commercial baseline Torque Seal with the HAP-free version to assist in meeting NESHAP requirements and sustainability goals.

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List of Symbols, Abbreviations, and Acronyms

ARL	U.S. Army Research Laboratory
ASTM	American Society for Testing and Materials
EPA	Environmental Protection Agency
Eta	(η)-viscosity
Gamma	(γ)-shear rate
HAP	Hazardous air pollutant
Kappa	(κ)-flow consistency index
mW	Milliwatt
n	Flow behavior index
NDCEE	National Defense Center for Energy and Environment
NESHAP	National Emission Standard for Hazardous Air Pollutants
Pa*s	Pascal second
R^2	Coefficient of determination
SPOTA	Sustainable Painting Operations for the Total Army
T_g	Glass transition temperature

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